

AN 1997-150014 [14] WPIDS
DNC C1997-048212
TI Aluminium (alloy) sputtering target - has inclusions
present at sputtering surface under inclusions, and specified
amount of oxygen.
DC M13
PA (NIKK-N) NIKKO GOLD FOIL CO LTD; (NIHA) JAPAN ENERGY CORP
CYC 1
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PRAI JP 1995-192619 19950706
AB JP 09025564 A UPAB: 19970407
High purity Al(alloy) sputtering target has inclusions
of at least 10 μ m in mean size to be present at the sputtering
surface of under 40 inclusions/cm², and oxygen content of under
15 ppm.
USE - For lessening particles upon forming thin film by sputtering.
Dwg.0/0

PATENT ABSTRACTS OF JAPAN

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(71)Applicant : JAPAN ENERGY CORP

(22)Date of filing : 06.07.1995

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NAGASAWA TAKASHI

OKABE GAKUO

(54) ALUMINUM OR ALUMINUM ALLOY SPUTTERING TARGET

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a high purity aluminum or aluminum alloy sputtering target small in the generation of particles.

SOLUTION: In a high purity aluminum (alloy) sputtering target, particles at the time of sputtering are generated by the burst of inclusions, particularly, oxides in the target, furthermore, the resticking of the grains to the vicinity of pores opened by the burst occurs, and this restuck materials peel to cause particles. Then, the abundance of inclusions with ≥ 10 μm average diameter appearing on the sputtering face of the target is regulated to < 40 pieces/cm², and furthermore, the content of oxygen in the target is regulated to < 150 ppm.

LEGAL STATUS

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CLAIMS

[Claim(s)]

[Claim 1] The abundance of the inclusion with an average diameter of 10 micrometers or more which appears in the spatter side of this target in high grade aluminum or a high grade aluminium alloy sputtering target is 2 40 pieces/cm. The aluminum or the aluminium alloy sputtering target characterized by being the following and the oxygen content in this target being less than 15 ppm further.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] About high grade aluminum or a high grade aluminium alloy sputtering target, in case this invention forms a thin film by sputtering especially, it relates to high grade aluminum with little generating of particle, or a high grade aluminium alloy sputtering target.

[0002]

[Description of the Prior Art] A sputtering target is a usually disc-like plate used as the source of sputtering for forming the electrode of various semiconductor devices, the gate, wiring, a component, an insulator layer, a protective coat, etc. on a substrate by sputtering. When the accelerated particle collides with a target front face, it deposits on the substrate with which the atom which constitutes a target by exchange of momentum is emitted to space, and counters. As a sputtering target, aluminum and an aluminium alloy target, a refractory metal and an alloy (the alloy like W-Ti, such as W, Mo, Ti, Ta, Zr, and Nb) target, metal silicide targets (MoSiX, WSix, NiSix, etc.), a platinum metal target, etc. have been used typically.

[0003] One of the important things also in such a target is the aluminum and the aluminium alloy target for aluminum wiring formation. The aluminum thin film is used also for the reflector of a compact disk or a magneto-optic disk again.

[0004]

[Problem(s) to be Solved by the Invention] Wiring width of face of a circuit is being made detailed with 1 micrometer or less with high integration of LSI. Thus, the particle in the case of the thin film formation by sputtering has been a big problem as an open circuit of a circuit, or a cause of a short circuit. It adheres to the thin film on a substrate directly, or the particle which disperses from a target on the occasion of sputtering carries out clustering, exfoliates after adhesion / deposition on a perimeter wall or components, and "particle" adheres on a thin film. also in an aluminum (alloy) target, particle is decreased with quality improvement of the reflector of detailed-izing and the compact disk of aluminum wiring, or a magneto-optic disk -- especially -- the number of particle with a dimension of 0.5 micrometers or more -- 50 piece/cm² To consider as the following is wished.

[0005] Under such a situation, when this invention formed a thin film by sputtering, it made it the technical problem to offer high grade aluminum with little generating of particle, or a high grade aluminium alloy sputtering target.

[0006]

[Means for Solving the Problem] In order that this invention persons may solve the above troubles, as a result of trying hard wholeheartedly, the reattachment of a particle arose near the hole opened by generating when the inclusion in a target explodes, and burst, and the particle in the case of sputtering acquired knowledge that this reattachment object exfoliates and this also causes particle. Therefore, in order to decrease such particle, it can respond by reducing the inclusion in a target as much as possible. It is especially inclusion frequency with an average diameter of 10 micrometers or more 1 cm² It became clear by considering as less than 40 hits that generating of particle could be lessened enough.

Moreover, it also became clear that it was what the inclusion leading to particle becomes mainly from an oxide, it assumed that all the oxygen in a target became the aluminum-oxide particle which is the diameter of 10 micrometers, and what the oxygen content in a target should be made less than 15 ppm also for was found out.

[0007] For this invention, based on these knowledge, the abundance of the inclusion with an average diameter of 10 micrometers or more which appears in the spatter side of this target in high grade aluminum or a high grade aluminium alloy sputtering target is 2 40 pieces/cm. It is the following and the aluminum or the aluminium alloy sputtering target characterized by the oxygen content in this target being less than 15 ppm further is offered.

[0008]

[Function] The particle in the case of sputtering is generated when the inclusion in a target explodes. The reattachment of a particle arises near the hole furthermore opened by burst, this reattachment object exfoliates, and this also causes particle. It is inclusion frequency with an average diameter of 10 micrometers or more 1cm 2 Generating of particle can be enough lessened by considering as less than 40 hits. Moreover, since the inclusion leading to particle mainly consists of an oxide, the oxygen content in a target is set to less than 15 ppm.

[0009]

[Detailed Description of the Invention] The high grade aluminum used as a material of the sputtering target of this invention means aluminum 4Ns or more, and an aluminium alloy contains a kind or two sorts or more for elements, such as Si, Cu, Ti, germanium, Cr, Mo, etc. which are usually added as a sputtering target, 10 or less % of the weight to high grade aluminum. Moreover, as a raw material used for manufacture of the sputtering target of this invention, although a commercial high grade aluminum ingredient and the above-mentioned alloy addition component ingredient can be used, what reduced impurity contents which have a bad influence on an electron device etc., such as a radioactive element and alkali metal, as much as possible is desirable.

[0010] A target dissolves and casts a raw material, it usually performs heat treatment and processing processing in order to make the material after casting suitable for the crystalline structure, particle size, etc., and it is produced by making the last target dimensions, such as discoid, after that. Quality, such as crystal orientation of a target, can be adjusted by combining plastic working, such as rolling and forging, and heat treatment appropriately.

[0011] Although inclusion is generated mainly in process of the dissolution of a raw material and casting and it is an oxide, a nitride, carbide, a hydride, a sulfide, silicide, etc., since it mainly consists of an oxide, the crucible used in case a target is manufactured, the gate, mold, etc. are good to use the thing of the ingredient made from graphite with reducibility, for example, a product. Moreover, before performing casting of the fused aluminum or the aluminium alloy, it is necessary to fully remove slags, such as an oxide which floats on the front face of the fused metal. The dissolution and casting are preferably performed in a vacuum among a non-oxidizing atmosphere.

[0012] The particle in the case of sputtering is generated when the inclusion in a target explodes. The reattachment of a particle arises near the hole furthermore opened by burst, this reattachment object exfoliates, and this also causes particle. The average diameter is larger than 10 micrometers, especially inclusion frequency with an average diameter of 10 micrometers or more sets to the spatter side of a target, and the inclusion leading to [of particle / main] generating is 2 40 pieces/cm. If it becomes above, especially generating of particle will increase. Therefore, the number of the inclusion with an average diameter of 10 micrometers or more which appears in the spatter side of the manufactured aluminum or an aluminium alloy sputtering target is 2 40 pieces/cm. It is made to become the following.

[0013] In addition, although the inclusion number which appears in the spatter side of a target is measured by microscope observation of the spatter side of a target etc., in the case of an alloy target etc., inclusion may necessarily be unable to grasp it clearly. In such a case, it can assume that all the oxygen in a target becomes an oxide, it can convert from the measured value of an oxygen content, and the number of inclusion can be presumed. When it is assumed that all the oxygen in a target becomes the

aluminum-oxide particle which is the diameter of 10 micrometers, the number of inclusion is 2 40 pieces/cm. An oxygen content is set to less than 15 ppm in order to be the following. Therefore, it is necessary to set the oxygen content in a target to less than 15 ppm. Since inclusion mainly consists of an oxide, if an oxygen content increases, according to it, the inclusion in a target which mainly consists of an oxide will increase, and the yield of the particle at the time of being sputtering will increase.

[0014] In this way, it is the number of particle with a dimension of 0.5 micrometers or more 50 pieces/cm by this invention 2 It can consider as the following and can respond to a demand at a future aluminum (alloy) target.

[0015]

[Example]

(Example 1) the inclusion number with an average diameter of 10 micrometers or more -- 21 piece/cm² it is -- the spatter membrane formation trial was performed using the high grade aluminum target whose oxygen content in a target is 8 ppm. The particle on a substrate was observed with the optical microscope, and the number was counted. consequently, the number of particle of 0.5 micrometers or more -- 32 piece/cm² it was .

[0016] (Example 1 of a comparison) the inclusion number with an average diameter of 10 micrometers or more -- 82 piece/cm² it is -- the spatter membrane formation trial was performed using the high grade aluminum target whose oxygen content in a target is 30 ppm. The particle on a substrate was observed with the optical microscope, and the number was counted. consequently, the number of particle of 0.5 micrometers or more -- 160 piece/cm² it was .

[0017] (Example 2) the inclusion number with an average diameter of 10 micrometers or more -- 11 piece/cm² it is -- the spatter membrane formation trial was performed using the aluminum-Cu0.5wt% alloy target whose oxygen content in a target is 4 ppm. The particle on a substrate was observed with the optical microscope, and the number was counted. consequently, the number of particle of 0.5 micrometers or more -- 18 piece/cm² it was .

[0018] (Example 2 of a comparison) the inclusion number with an average diameter of 10 micrometers or more -- 63 piece/cm² it is -- the spatter membrane formation trial was performed using the aluminum-Cu0.5wt% alloy target whose oxygen content in a target is 23 ppm. The particle on a substrate was observed with the optical microscope, and the number was counted. Consequently, the 98 numbers /of particle of 0.5 micrometers or more were [cm] 2.

[0019]

[Effect of the Invention] By using the high grade aluminum or the high grade aluminium alloy target of this invention, particle generating in the case of sputtering can be reduced. Especially, it is the number of particle with a dimension of 0.5 micrometers or more 50 pieces/cm² It can consider as the following.

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TECHNICAL FIELD

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PRIOR ART

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EFFECT OF THE INVENTION

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] Wiring width of face of a circuit is being made detailed with 1 micrometer or less with high integration of LSI. Thus, the particle in the case of the thin film formation by sputtering has been a big problem as an open circuit of a circuit, or a cause of a short circuit. It adheres to the thin film on a substrate directly, or the particle which disperses from a target on the occasion of sputtering carries out clustering, exfoliates after adhesion / deposition on a perimeter wall or components, and "particle" adheres on a thin film. also in an aluminum (alloy) target, particle is decreased with quality improvement of the reflector of detailed-izing and the compact disk of aluminum wiring, or a magneto-optic disk -- especially -- the number of particle with a dimension of 0.5 micrometers or more -- 50 piece/cm² To consider as the following is wished.

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MEANS

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OPERATION

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EXAMPLE

[Example]

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[0018] (Example 2 of a comparison) the inclusion number with an average diameter of 10 micrometers or more -- 63 piece/cm² it is -- the spatter membrane formation trial was performed using the aluminum-Cu0.5wt% alloy target whose oxygen content in a target is 23 ppm. The particle on a substrate was observed with the optical microscope, and the number was counted. Consequently, the 98 numbers /of particle of 0.5 micrometers or more were [cm] 2.

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(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開平9-25564

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審査請求 未請求 請求項の数 1 FD (全 3 頁)

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(22) 出願日	平成7年(1995)7月6日	(72) 発明者	福世 秀秋 茨城県北茨城市華川町白堀187番地4 株式 会社ジャパンエナジー磯原工場内
		(72) 発明者	永澤 俊 茨城県北茨城市華川町白堀187番地4 株式 会社ジャパンエナジー磯原工場内
		(72) 発明者	岡部 岳夫 茨城県北茨城市華川町白堀187番地4 株式 会社ジャパンエナジー磯原工場内
		(74) 代理人	弁理士 倉内 基弘 (外1名)

(54) 【発明の名称】 アルミニウムまたはアルミニウム合金スパッタリングターゲット

(57) 【要約】

【目的】 パーティクルの発生が少ない高純度アルミニウムまたはアルミニウム合金スパッタリングターゲットの開発。

【構成】 高純度アルミニウム(合金)スパッタリングターゲットにおいて、スパッタリングの際のパーティクルは、ターゲット中の介在物、特に酸化物が破裂することにより、さらに破裂により開いた穴の近傍に粒子的再付着が生じ、この再付着物が剥離してパーティクルの原因となる。そこで、ターゲットのスパッタ面に現れる平均直径10μm以上の介在物の存在量を40個/cm²未満、さらにターゲット中の酸素含有量を15ppm未満とする。

(2)

特開平9-25564

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【特許請求の範囲】

【請求項1】 高純度アルミニウムまたは高純度アルミニウム合金スパッタリングターゲットにおいて、該ターゲットのスパッタ面に現れる平均直径 $10\mu\text{m}$ 以上の介在物の存在量が $40\text{個}/\text{cm}^2$ 未満であり、さらに該ターゲット中の酸素含有量が 15ppm 未満であることを特徴とするアルミニウムまたはアルミニウム合金スパッタリングターゲット。

【発明の詳細な説明】

【0001】

【産業上の利用分野】 本発明は、高純度アルミニウムまたは高純度アルミニウム合金スパッタリングターゲットに関するものであり、特にスパッタリングにより薄膜を形成する際にパーティクルの発生が少ない高純度アルミニウムまたは高純度アルミニウム合金スパッタリングターゲットに関するものである。

【0002】

【従来の技術】 スパッタリングターゲットは、スパッタリングにより各種半導体デバイスの電極、ゲート、配線、素子、絶縁膜、保護膜等を基板上に形成するためのスパッタリング源となる。通常は円盤状の板である。加速された粒子がターゲット表面に衝突するとき運動量の交換によりターゲットを構成する原子が空間に放出されて対向する基板上に堆積する。スパッタリングターゲットとしては、アルミニウム及びアルミニウム合金ターゲット、高融点金属及び合金（W、Mo、Ti、Ta、Zr、Nb）等及びW-Tiのようなその合金）ターゲット、金属シリサイド（ MoSi_2 、 WSi_2 、 NiSi 、等）ターゲット、白金族金属ターゲット等が代表的に使用されてきた。

【0003】 こうしたターゲットの中でも重要なものの一つが、アルミニウム配線形成用のアルミニウム及びアルミニウム合金ターゲットである。アルミニウム薄膜はまたコンパクトディスクや光磁気ディスクの反射面にも使用されている。

【0004】

【発明が解決しようとする課題】 LSIの高集積化に伴い、回路の配線幅は $1\mu\text{m}$ 以下と微細化されつつある。このような中でスパッタリングによる薄膜形成の際のパーティクルが、回路の断線あるいは短絡の原因として大きな問題になっている。「パーティクル」とは、スパッタリングの際にターゲットから飛散する粒子がクラスター化して基板上の薄膜に直接付着したり、或いは周囲壁や部品に付着・堆積後剥離して薄膜上に付着するものである。アルミニウム（合金）ターゲットにおいても、アルミニウム配線の微細化やコンパクトディスクや光磁気ディスクの反射面の高品質化に伴い、パーティクルを減少させる、特に $0.5\mu\text{m}$ 以上の寸法のパーティクルの数を $50\text{個}/\text{cm}^2$ 以下とすることが所望される。

【0005】 こうした状況の下で、本発明は、スパッタ

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リングにより薄膜を形成する際にパーティクルの発生が少ない高純度アルミニウムまたは高純度アルミニウム合金スパッタリングターゲットを提供することを課題とした。

【0006】

【課題を解決するための手段】 本発明者らは、上記のような問題点を解決するために鋭意努力した結果、スパッタリングの際のパーティクルは、ターゲット中の介在物が破裂することにより発生すること、また破裂により開いた穴の近傍に粒子の再付着が生じ、この再付着物が剥離してこれもまたパーティクルの原因となるとの知見を得た。従って、これらのパーティクルを減少させるためには、ターゲット中の介在物を極力減らすことにより対応が可能である。特に平均直径 $10\mu\text{m}$ 以上の介在物頻度を 1cm^2 当たり 40個 未満とすることによりパーティクルの発生を十分少なくすることができることが判明した。また、パーティクルの原因となる介在物が主として酸化物からなるものであることも判明し、ターゲット中の酸素が全て、直径 $10\mu\text{m}$ の酸化アルミニウム粒子になると仮定して、ターゲット中の酸素含有量を 15ppm 未満とすべきことも見いだされた。

【0007】 これら知見に基づいて、本発明は、高純度アルミニウムまたは高純度アルミニウム合金スパッタリングターゲットにおいて、該ターゲットのスパッタ面に現れる平均直径 $10\mu\text{m}$ 以上の介在物の存在量が $40\text{個}/\text{cm}^2$ 未満であり、さらに該ターゲット中の酸素含有量が 15ppm 未満であることを特徴とするアルミニウムまたはアルミニウム合金スパッタリングターゲットを提供するものである。

【0008】

【作用】 スパッタリングの際のパーティクルは、ターゲット中の介在物が破裂することにより発生する。さらに破裂により開いた穴の近傍に粒子の再付着が生じ、この再付着物が剥離してこれもまたパーティクルの原因となる。平均直径 $10\mu\text{m}$ 以上の介在物頻度を 1cm^2 当たり 40個 未満とすることによりパーティクルの発生を十分少なくすることができる。また、パーティクルの原因となる介在物が主として酸化物からなるものであるから、ターゲット中の酸素含有量を 15ppm 未満とする。

【0009】

【発明の具体的な説明】 本発明のスパッタリングターゲットの素材として用いる高純度アルミニウムとは 4N 以上のアルミニウムを意味し、アルミニウム合金とはスパッタリングターゲットとして通常添加されるSi、Cu、Ti、Ge、Cr、Mo等の元素を高純度アルミニウムに一種または二種以上を $10\text{重量}\%$ 以下含有するものである。また、本発明のスパッタリングターゲットの製造に用いる原料としては、市販の高純度アルミニウム材料および上記の合金添加成分材料を使用することがで

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さるが、電子デバイス等に悪影響を及ぼす放射性元素、アルカリ金属等の不純物含有量を極力低減したものが好ましい。

【0010】ターゲットは通常、原料を溶解及び铸造し、铸造後の素材を、結晶組織、粒徑等を適切なものとするため熱処理および加工処理を施し、その後、円盤状等の最終ターゲット寸法に仕上げることににより作製される。圧延や鍛造等の塑性加工と熱処理を適切に組み合わせることによりターゲットの結晶方位等の品質の調整を行なうことができる。

【0011】介在物は、主として原料の溶解、铸造の過程で発生し、酸化物、窒化物、炭化物、水素化物、硫化物、珪化物などであるが、主として酸化物からなるものであることから、ターゲットを製造する際に使用するルツボ、揚口、モールドなどは還元性のある材料、例えばグラファイト製のものを使用するのがよい。また、溶解したアルミニウムまたはアルミニウム合金の铸造を行う前に、溶解した金属の表面に浮かんてくる酸化物等のスラグを十分に除去する必要がある。溶解、铸造は非酸化性雰囲気中、好ましくは真空中で行う。

【0012】スパッタリングの際のパーティクルは、ターゲット中の介在物が破裂することにより発生する。さらに破裂により開いた穴の近傍に粒子の再付着が生じ、この再付着物が剥離してこれもまたパーティクルの原因となる。パーティクルの主な発生原因となる介在物はその平均直径が $10\mu\text{m}$ よりも大きいものであり、特に平均直径 $10\mu\text{m}$ 以上の介在物濃度が、ターゲットのスパッタ面において $40\text{個}/\text{cm}^2$ 以上になると、特にパーティクルの発生が多くなる。従って、製造したアルミニウムまたはアルミニウム合金スパッタリングターゲットのスパッタ面に現れる平均直径 $10\mu\text{m}$ 以上の介在物の個数は $40\text{個}/\text{cm}^2$ 未満になるようにする。

【0013】なお、ターゲットのスパッタ面に現れる介在物個数は、ターゲットのスパッタ面の顕微鏡観察などにより測定されるが、合金ターゲット等の場合には必ずしも介在物が明瞭に把握できない場合がある。このような場合にはターゲット中の酸素が全て酸化物になると仮定し、酸素含有量の測定値から換算して介在物の個数を推定することができる。ターゲット中の酸素が全て、直径 $10\mu\text{m}$ の酸化アルミニウム粒子になると仮定した場合に、介在物の個数が $40\text{個}/\text{cm}^2$ 未満であるためには、酸素含有量は 15ppm 未満となる。従って、ターゲット中の酸素含有量は 15ppm 未満にする必要があ

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る。介在物が主に酸化物からなることから、酸素含有量が増えると、それに応じてターゲット中の主として酸化物からなる介在物が増加し、スパッタリングの際のパーティクルの発生量が増える。

【0014】こうして、本発明により、 $0.5\mu\text{m}$ 以上の寸法のパーティクルの数を $50\text{個}/\text{cm}^2$ 以下とすることができ、今後のアルミニウム（合金）ターゲットへの要求に対応することができる。

【0015】

10 【実施例】

（実施例1）平均直径 $10\mu\text{m}$ 以上の介在物個数が $21\text{個}/\text{cm}^2$ であり、ターゲット中の酸素含有量が 8ppm である高純度アルミニウムターゲットを用いてスパッタ成膜試験を行った。基板上的パーティクルを光学顕微鏡で観察し個数を数えた。その結果、 $0.5\mu\text{m}$ 以上のパーティクル数は $32\text{個}/\text{cm}^2$ であった。

【0016】（比較例1）平均直径 $10\mu\text{m}$ 以上の介在物個数が $82\text{個}/\text{cm}^2$ であり、ターゲット中の酸素含有量が 30ppm である高純度アルミニウムターゲットを用いてスパッタ成膜試験を行った。基板上的パーティクルを光学顕微鏡で観察し個数を数えた。その結果、 $0.5\mu\text{m}$ 以上のパーティクル数は $160\text{個}/\text{cm}^2$ であった。

【0017】（実施例2）平均直径 $10\mu\text{m}$ 以上の介在物個数が $11\text{個}/\text{cm}^2$ であり、ターゲット中の酸素含有量が 4ppm であるアルミニウム-Cu $0.5\text{wt}\%$ 合金ターゲットを用いてスパッタ成膜試験を行った。基板上的パーティクルを光学顕微鏡で観察し個数を数えた。その結果、 $0.5\mu\text{m}$ 以上のパーティクル数は $18\text{個}/\text{cm}^2$ であった。

30 【0018】（比較例2）平均直径 $10\mu\text{m}$ 以上の介在物個数が $63\text{個}/\text{cm}^2$ であり、ターゲット中の酸素含有量が 23ppm であるアルミニウム-Cu $0.5\text{wt}\%$ 合金ターゲットを用いてスパッタ成膜試験を行った。基板上的パーティクルを光学顕微鏡で観察し個数を数えた。その結果、 $0.5\mu\text{m}$ 以上のパーティクル数は $98\text{個}/\text{cm}^2$ であった。

【0019】

40 【発明の効果】本発明の高純度アルミニウムまたは高純度アルミニウム合金ターゲットを用いることにより、スパッタリングの際のパーティクル発生を低減することができる。特に、 $0.5\mu\text{m}$ 以上の寸法のパーティクルの数を $50\text{個}/\text{cm}^2$ 以下とすることができる。